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# Influence of different treatments of the ceramic surface and thermal cycling on the bond strength of brackets to ceramic

*Influência de diferentes tratamentos de superfície da cerâmica e ciclagem térmica na resistência de união de bráquetes à cerâmica*

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## Resumo

**Objetivo:** Avaliar *in vitro* o efeito de diferentes tratamentos de superfície da cerâmica e ciclagem térmica na resistência da união ao cisalhamento (RUC) de bráquetes metálicos colados na cerâmica felspática. **Material e método:** Cilindros cerâmicos foram separados em 4 grupos (n=4) de acordo com os tratamentos da superfície da cerâmica: G1-Clearfil Ceramic Primer silano e Transbond XT (CCPT); G-condicionamento com ácido hidrofluorídrico a 10% (AHF) por 60s, CCP e Transbond XT (ACCPT); G3-condicionamento com AHF a 10% por 60s, Adesivo Ambar e Transbond XT (AAAT); e, G4 condicionamento com AHF a 10% por 60s, RelyX Ceramic Primer silano, *primer* adesivo Transbond e Transbond XT (ACPAT). Os bráquetes foram fixados nos cilindros com Transbond XT e fotoativado por 40s com LED Radii Plus. Todas as amostras foram armazenadas em água deionizada a 37 °C por 24 h e dois cilindros de cada grupo foram submetidos a 7.000 ciclos térmicos na máquina para ciclagem térmica (5 °C/55 °C). Após armazenagem e ciclagem térmica, a RUC foi realizada à velocidade de 1 mm/min. Na Análise de Variância de 2 fatores e ao teste de Tukey's post hoc test (α=0,05) a RUC do G2 foi significante maior do que dos demais grupos (p<0,05). **Resultado:** As amostras submetidas à ciclagem térmica apresentaram valores de RUC significantemente menores do que as amostras sem ciclagem térmica (p<0,05), independente do tratamento de superfície da cerâmica. **Conclusão:** Nas condições desse estudo o melhor resultado para colagem foi obtido com o condicionamento, silano CCP e Transbond XT. A ciclagem térmica reduziu a RUC em todos os grupos.

**Descritores:** Resistência de união; bráquetes ortodônticos; silano; adesivo; cerâmica.

## Abstract

**Objective:** To evaluate *in vitro* the effect of different treatments of the ceramic surface and thermal cycling on the shear bond strength (SBS) of metallic brackets bonded to feldspathic ceramic. **Material and method:** Ceramic cylinders were divided into four groups (n=4) according to the treatment of ceramic surface: G1-Clearfil Ceramic Primer silane and Transbond XT (CCPT); G2-etched with 10% hydrofluoric acid (HFA) for 60 s, CCP and Transbond XT (ACCPT); G3-etched with 10% HFA for 60 s, Ambar Adhesive and Transbond XT (AAAT); and, G4 - etched with 10% HFA for 60 s, RelyX Ceramic Primer silane -RCP, adhesive primer Transbond and Transbond XT (ACPPT). Brackets were bonded to the cylinders with Transbond XT and light-activated for 40 s with LED Radii Plus. All specimens were stored in deionized water at 37 °C for 24 h, and two cylinders from each group were subject to 7,000 thermal cycles in a thermal cycler (5 °C/55 °C). After storage and thermal cycling, the SBS test was performed at a crosshead speed of 1 mm/min. Data were subjected to two-way ANOVA and Tukey's post hoc test (α=0.05). **Result:** The SBS of ACCPT was significantly higher than the other groups (p<0.05). The specimens submitted to thermal cycling showed significantly lower SBS than those without thermal cycling (p<0.05), regardless the ceramic surface treatment. The ARI showed predominance of score 0 for all groups. **Conclusion:** Acid etching, CCP silane and Transbond XT method obtained the best results for bracket bonding. Thermal cycling reduced SBS for all groups. Score 0 was predominant for ARI in all groups.

**Descriptors:** Shear strength; orthodontic brackets; silane; adhesive; ceramic.

## INTRODUCTION

In the last years, older patients have looking for orthodontic treatment and many of these have ceramic restorations. Ceramics have gained great notoriety due to markable improvement of the ability to mimic dental tissues, mechanical strength and this material is the most commonly used for indirect restorative in Dentistry[1]. However, bonding orthodontic brackets to ceramic surface needs special care because they can exhibit a higher degree of failure in relation to enamel bond[2]. On the other hand, some studies showed no negative effects on ceramic surface, since the most failures was adhesive between the bonding material and the ceramic surface[3-5].

Normally, hydrofluoric acid (HF) has been used in orthodontics for etching ceramic surfaces for 60 s, where the orthodontic bracket is bonded[3]. HF acid is an efficient surface modification agent that is capable of dissolving the glassy ceramic matrix, increased the roughness[6], contact surface and wettability[7,8], promoting higher bond between bonding materials and ceramics[9-11]. The application of hydrofluoric acid followed by a silane solution have been used prior to bonding glass ceramic to increase the bond strength and durability between brackets and ceramic surface[3,12,13] or luting glass ceramic restorations[12].

Although, HF acid is well accepted by dental professionals because the solution is easy and quick to apply, effective and cheap[14]. However, it may be harmful for patients and dental personnel[10] because it is corrosive, highly toxic and reactive[15]. Thereby, a primer adhesive Clearfil Ceramic Primer (CCP, Kuraray America) specific for ceramics has been indicated without the need of HF acid etching. A study showed that CCP silane could be used without previous acid etching of the ceramic[2]. On the other hand, it is questionable if the resin cement and silane are effective in wetting the ceramic surface and filling up irregularities when HF acid is not used[9]. Previous study showed that the use of adhesive improves adaptation and bond strength of substrates along the resin cement/ceramic interface[9].

After bonded to ceramic surface, the orthodontic brackets are exposed to the oral environment and failure may occur at interface among orthodontic brackets, bonding material and dental ceramic due to thermal changes and heavy forces produced by the archwire in the oral cavity[4]. The literature is still not conclusive about thermal cycling protocols. Temperature between 500 and 7,000 cycles have been used to verify if temperature changes promoted stresses at the interface causing deteriorations in simulated oral conditions, before the mechanical test[4,16].

The purpose of this study was to evaluate the effect of different treatments and thermal cycling on the SBS of metallic brackets bonded to feldspathic ceramic. The hypotheses tested were: 1) Different treatments on the ceramic surface would not affect the shear bond strength; and 2) Thermal cycling would not affect the shear bond strength.

## MATERIAL AND METHOD

### *Preparation of the Specimens and Light-curing Procedures*

Sixteen feldspathic ceramic glazed cylinders with 20 mm in height x 13 mm in diameter (Certec Advanced Ceramics, Barueri, SP, Brazil) were used in this study. The surface of ceramic was cleaned using a rubber cup (KG Sorensen, Cotia, SP, Brazil) and pumice-water slurry (S.S. White, Petropolis, RJ, Brazil) for 30 s, rinsed with air-water spray for 30 s and dried with air for 30 The rubber cup was replaced after each cylinder.

The sixteen cylinders were randomly assigned into four groups (n=4) according to the surface treatments: G1 - Clearfil Ceramic Primer silane (Kuraray Medical Inc., Kurashiki, Okayama, Japan - CCP) and Transbond XT (3M Unitek, Monrovia, CO, USA) - CCPT; G2 - etched with 10% hydrofluoric acid (HFA) gel for 60 s (Dentsply Caulk, Milford, DE, USA), rinsed with air-water spray for 30 s and dried with air for 30 s, CCP (Kuraray) and Transbond XT (3M Unitek) - ACCPT; G3 - etched with 10% HFA gel for 60 s (Dentsply Caulk), rinsed with air-water spray for 30 s and dried with air for 30 s, Ambar Universal Adhesive (FGM, Joinville, SC, Brasil) and Transbond XT (3M Unitek) - AAAT; and, G4 - etched with 10% HFA gel for 60 s (Dentsply Caulk), rinsed with air-water spray for 30 s and dried with air for 30 s, RelyX Ceramic Primer silane - RCP (3M ESPE, Saint Paul, MN, EUA), adhesive primer (3M Unitek, Monrovia, CO, USA) and Transbond XT (3M UNITEK) - ARCPPT.

In group 1, two layers of CCP silane (Kuraray) were applied on the ceramic surface and air dried for 60 s. Stainless steel standard maxillary premolar brackets (Morelli, Sorocaba, SP, Brazil) were bonded and seated firmly to the curved area of the ceramic cylinders using Transbond XT (3M Unitek) following the manufacturer's instructions. Bonding resin excess was removed using microbrushes, and light-activation was carried out with 4 exposures (10 s), one on each side of the bracket for a total exposure time of 40 s using LED device (Radii Plus; SDI Limited, Bayswater, Victoria, Australian) having an irradiance of 1,100 mW/cm$^2$, measured by power meter (Ophir Optronics Inc., Danvers, MA, USA) and computer-controlled spectrometer (USB2000; Ocean Optics, Dunedin, FL, USA).

In group 2, after etched with 10% HFA gel for 60 s (Dentsply), rinsed and dried, two layers of CCP silane (Kuraray) were applied on the ceramic surface and air dried for 60 s. The bonding procedure of the brackets with the Transbond XT was similar to group 1.

In group 3, after etched with 10% HFA gel for 60 s (Dentsply), rinsed and dried, one layer of a light cured Ambar Universal Adhesive (FGM) were applied on the etched area of ceramic surface and exposed a LED (Radii Plus, SDI) for 20 s. The bonding procedure of the brackets with the Transbond XT was similar to group 1.

In group 4, after etched with 10% HFA gel for 60 s (Dentsply), rinsed and dried, two layers of RCP silane (3M ESPE) were applied on the ceramic surface and air dried for 60 s. One layer of light cured adhesive primer (3M Unitek) were applied on the etched area of ceramic surface after silane and exposed to LED (Radii Plus, SDI) for 20 s. The bonding procedure of the brackets with the Transbond XT was similar to group 1.

### *Storage and Thermal Cycling*

After the bonding procedures, all specimens were stored in deionized water at 37 °C for 24 h. Afterwards, two cylinders from each group were subject to 7,000 thermal cycles in a thermal cycler (MSCT 3; Marnucci ME, Sao Carlos, SP, Brazil) with deionized water between 5 °C and 55 °C (dwell time of 30 s), and transfer time of 10 s between baths. Five metallic brackets were bonded to each ceramic cylinder (n=5) for each surface treatment and thermal cycling, totalized 80 bonded brackets.

### *Shear Bonding Strength (SBS)*

After thermal cycling, a mounting jig was used to align the ceramic-bracket interface parallel to the testing device. The SBS was performed in an universal mechanical testing machine (Model 4411; Instron, Canton, MA, USA) with a knife-edged rod at a crosshead speed of 1.0 mm/min until failure occurs. The SBS data were calculated in MPa and subject to two-way ANOVA and Tukey's post hoc test (α=0.05).

### *Failure Analysis*

After debonding, the bracket and ceramic surfaces were observed under optical microscopy (Olympus Corp, Tokyo, Japan) at 40× magnification. The Adhesive Remaining Index (ARI) was used to classify the failure modes[17] as follows: Score 0: no bonding resin on the ceramic; Score 1: less than half of the bonding resin remained on the ceramic; Score 2: half of the bonding resin was left on the ceramic; and, Score 3: all bonding resin was left on the ceramic, with clear impression of the bracket mesh.

## RESULT

The SBS mean values are shown in Tables 1 and 2. Significant influences of treatment ($p<0.00001$) and thermal cycling ($p<0.00001$) were detected. The interaction between treatment and thermal cycling ($p=0.55708$) factors were not significant.

**Table 1.** Mean shear strength values (S.D.) in MPa for treatment, regardless of thermal cycling

| Treatment | Shear bond strength (MPa) |
|---|---|
| Group 2 - Acid + Clearfil Ceramic Primer + Transbond XT | 9.8 ± 0.4 a |
| Group 4 - Acid + Rely Ceramic Primer + Primer Transb + Transbond XT (ARCPPT) | 8.3 ± 0.5 b |
| Group 1 - Clearfil Ceramic Primer + Transbond XT (CCPT) | 8.1 ± 0.7 b |
| Group 3 - Acid + Adhesive Ambar + Transbond XT (AAAT) | 5.5 ± 0.3 c |

Means followed by different lowercase letters in the column indicate statistically significant difference (p<0.05).

**Table 2.** Mean shear strength values (S.D.) in MPa with or without thermal cycling, egardless of treatment

| Thermal cycling | Shear bond strength (MPa) |
|---|---|
| Without | 9.6 ± 0.5 a |
| With | 6.4 ± 0.3 b |

Means followed by different lowercase letters in the column indicate statistically significant difference (p<0.05).

When treatment were compared (Table 1), the mean SBS of group 2 (ACCPT) was significantly higher than of group 4 (ARCPPT), group 1 (CCPT) and group 3 (AAAT) $p<0.05$. The group 4 (ARCPPT) and group 1 (CCPT) were significantly higher than of group 3 (AAAT) $p<0.05$. No significant differences ($p>0.05$) in bond strength were found between group 4 and group 1, regardless of thermal cycling. The specimens submitted to thermal cycling showed significantly lower SBS than those without thermal cycling ($p<0.05$), regardless of treatment (Table 2). Score 0 was predominant for ARI in all groups (Figure 1).

## DISCUSSION

Normally, ceramic surfaces are submitted to HF acid etching in order to promote alterations in the morphology and improve bond strength[18]. HF acid is indicated for ceramic that contains silica in the composition[19]. When in contact with the ceramic surface it dissolves the vitreous phase, exposing crystals, promoting the formation of microporosities on the surface of conditioned ceramics, increased surface area, promoting better contact between bonding materials and ceramic, and improved bonding quality[11,13,19].

The present study showed that the different treatments on the ceramic surface affect significantly the SBS, regardless thermal cycling. The results indicated that the first hypothesis was not accepted. When HF acid was used on the ceramic surface before other treatments, the results were more effective, except for group 3, when the Ambar Universal Adhesive was used. Some studies have shown that different treatments, such as phosphoric acid, aluminum oxide or without etching presented lower bond strength in relation to surface HF acid etching[2,11,13]. However, previous studies have shown that HF acid may promoted severe problems for dental personnel and patients because this solution is extremely toxic, reactive and corrosive[10,15], and the severity depends on the acid concentration, exposure time and penetration into the exposed

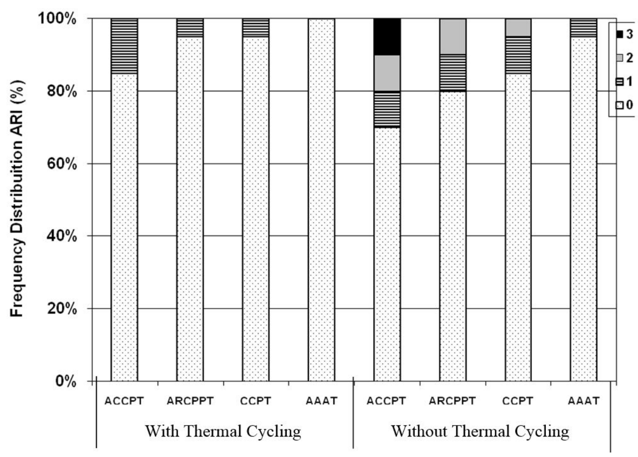


**Figure 1.** Frequency distributions (%) of the adhesive remnant index (ARI) scores for all groups. Score 0 indicates that no bonding resin remained on the ceramic; Score 1 indicates that less than half the bonding resin remained on the ceramic; Score 2 indicates that more than half of bonding resin remained on the ceramic; and, Score 3 indicates that all bonding resin was left on the ceramic, with a clear impression of the bracket mesh.

tissue[20]. Silane is applied on the ceramic surface after acid etching. The silanes are usually monomeric species where silicon links to reactive organic radicals and hydrolysable ester groups where the reactive organic groups bond to resin molecules and hydrolysable monovalent groups bond chemically to silicon in the glass matrix, consequently forming chemical bond between silane and silica layer on the ceramic surface or bond materials[13].

On the other hand, alternative methods have been proposed as CCP silane without prior acid etching. This silane contains MDP phosphate monomer in the composition that bonds strongly to metal oxides. In addition, it contains MPS silane coupling agent that promotes higher adhesion to esthetic restorative materials, such as dental ceramic[21]. A study showed that the mixture of silane and acidic monomers may accelerate the hydrolysis reaction of the alkoxy groups into silanol, increasing the reactivity of the silane[22]. However, in this study was observed that the groups acid etched showed higher bond strength than the groups with only silane, or acid and adhesive without silane. In the current study, CCP without acid etching showed bond strength value of 8.1 MPa higher than recommended by Reynolds in the range of 6 to 8 MPa[23].

Besides that, previous study showed that the effectiveness of bond when only silane is used depends on the ability of bonding materials to fill irregularities and promoted contact between the ceramic and bonding materials[9]. In this study, when an unfilled resin was used after RCP silane group 4, it infiltrated the etched surface irregularities; however, the bond strength was significantly lower than group 2 where after etching CCP silane was used before bonding materials, without unfilled resin. Probably, this fact occurred because this silane contains both MDP phosphate monomer that bonds strongly to metal oxides, and MPS silane that promotes higher adhesion to dental ceramic[21]. However, when the unfilled resin Ambar was used group 3 after acid etching without silane, the bond strength values (5.5 MPa) were significantly lower than other groups, and lower than that recommended in the literature[23]. Probably, this fact occurred because the silane has not been used after acid etching.

Another fact that can influence the bond strength between the ceramic surface and the bracket surface is the thermal cycling when bonding resin is used[3,16]. The thermal cycling test has been recommended and used to verify if temperature changes may influence the reduction of bond strength values between the ceramic surface, bonding materials and brackets[2]. Previous studies showed that the reduction of the bond strength could be due to continuous action of the water on the ceramic-bonding materials-brackets interface with hydrolytic degradation of the components[24]. In addition, different thermal expansion coefficients and abrupt temperature changes of bonding materials can produce thermal stresses at the interface and decrease the bond strength[24].

In this study, thermal cycling significantly decreased the SBS between bonding material and ceramic surface, whatever the treatment used. The results indicated that the second hypothesis was not accepted. The results of the present work are in agreement with those of previous studies, which found significant difference in bond strength values when the specimens were submitted to thermal cycling[2,4]. On the other hand, other studies are not in agreement showing no significant difference for bond strength of specimens submitted to thermal cycling[11,16]. These conflicting results may have occurred because a smaller number of thermal cycles were used in the aforementioned works in relation to a large number of thermal cycles in the current study. Another study showed that a larger number of cycles is indicate to obtain accelerated simulation of the bond degradation[16]. ARI values showed a predominance of debonding failures with score 0 (no bonding resin on the ceramic surface). Clinically, this fact may be advantageous because there is less bonding material to remove from the ceramic surface after bracket debonding[2-4].

In summary, the results of this study showed that acid etching before CCP silane influenced significantly the SBS. The thermal cycling significantly reduced the SBS values, whatever the treatment used. The CCP silane may be used without acid etching of the ceramic because showed SBS values than 6 MPa. However, care should be taken by clinicians when the adhesive is used without silane because it has not acceptable clinically SBS to resist forces during orthodontic treatment.

## CONCLUSION

It may be concluded that the HF acid, CCP silane and Transbond XT resin showed SBS significantly higher than other groups, regardless of thermal cycling. Thermal cycling significantly decreased the SBS, whatever the treatment used. Score 0 was predominant for ARI in all groups.

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## CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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